PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of	)	
	)	FOR: LOW PRESSURE REVERSE
John Tomaschke	)	OSMOSIS AND NANOFILTRA-
	)	TION MEMBRANES AND
Serial No.: 09/724,883	)	METHOD FOR PRODUCTION
	)	THEREOF
Filed: November 28, 2003	)	
	)	Group Art Unit: 1723

PROPOSED AMENDMENT

Commissioner for Patents
P. O. Box 2327
Arlington VA 22202

Attention: Krishnan S. Menon
Examiner

Dear Sir:

This is in response to the Office Action dated February 9, 2004. Please amend the above-identified application as follows:

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, Arlington VA 22202 on

April 2, 2004
(Mailing Date)

(Typed Name)

(Signature)

April 2, 2004
(Date of Signature)

IN THE CLAIMS

Amendments to the claims are indicated in comparison the last formal amendment filed on August 29, 2003.

1-14. (cancelled)

15. (thrice amended) A composite membrane useful for reverse osmosis or nanofiltration comprising:

a supportive porous under-structure; and

a top layer consisting of a crosslinked polyamide thin film which is adhered to the upper surface of the porous support structure, said top layer having been contacted with a solution of a C_1-C_6 alkyl, alkenyl, haloalkyl, haloalkenyl, or hydroxy ~~or aryl~~ sulfonic or disulfonic acid compound,

whereby said membrane has a water flux of at least about 15 gfd and a rejection of at least 20 percent when tested an a 0.05 percent aqueous sodium chloride at 75 psi and 25°C.

16. (once amended) A composite membrane as in Claim 15 wherein said crosslinked polyamide comprises the reaction product of an aromatic diamine or triamine and an aromatic triacyl halide.

17. (once amended) A composite membrane as in Claim 16 wherein said crosslinked polyamide comprises the reaction product of an aromatic diamine or triamine, an aromatic triacyl halide and an aromatic diacyl halide.

18. (once amended) A composite membrane as in Claim 15 wherein the porous support is

a polyarylethersulfone.

19. (once amended) A composite membrane as in Claim 15 wherein said first membrane comprises a thin film, flat sheet, hollow fiber or tubular membrane.

20. (once amended) A composite membrane as in Claim 15 wherein the membrane is a component of a spiral-wound membrane filter or a plate and frame filter.

21. (thrice amended) A composite membrane as in Claim 15 wherein said organic sulfonic or disulfonic acid compound comprises a sulfoacetic, sulfosuccinic~~hydroxybenzene sulfonic, methane sulfuonic~~ methanesulfonic, ethanedisulfonic, or hydroxybutane sulfonic, ~~benzenedisulfonic, dihydroxy benzene sulfonic or dihydroxy benzene disulfonic~~ compound or mixtures thereof.

22. (cancelled)

23. (twice amended) A composite membrane as in Claim 15 wherein said organic sulfonic acid compound comprises methanesulfonic acid, trifluoromethanesulfonic acid or a mixture thereof.

24. (cancelled)

25. (once amended) A composite membrane as in Claim 15 wherein said solution of an organic sulfonic acid compound comprises said organic sulfonic acid compound dispersed or dissolved in water, alcohol, glycol, alkoxy alcohol or a carboxylic acid or a mixture thereof.

26. (cancelled)

27. (new) A composite membrane useful for reverse osmosis or nanofiltration comprising:

a supportive porous under-structure; and

a top layer consisting of a crosslinked polyamide thin film which is adhered to the upper surface of the porous support structure, said top layer having been contacted with a solution of a C_1-C_6 alkyl, alkenyl, haloalkyl, haloalkenyl, or hydroxy sulfonic or disulfonic acid compound,

whereby said membrane has a water flux of at least 15 gfd and a rejection of at least 20 percent when tested an a 0.05 percent aqueous sodium chloride at 75 psi and 25°C.

28. (new) A composite membrane as in Claim 27 wherein said crosslinked polyamide comprises the reaction product of an aromatic diamine or triamine and an aromatic triacyl halide.

29. (new) A composite membrane as in Claim 28 wherein said crosslinked polyamide comprises the reaction product of an aromatic diamine or triamine, an aromatic triacyl halide and an aromatic diacyl halide.

30. (new) A composite membrane as in Claim 27 wherein the porous support is a polyarylethersulfone.

31. (new) A composite membrane as in Claim 27 wherein said first membrane comprises a thin film, flat sheet, hollow fiber or tubular membrane.

32. (new) A composite membrane as in Claim 27 wherein the membrane is a component

of a spiral-wound membrane filter or a plate and frame filter.

33. (new) A composite membrane as in Claim 27 wherein said organic sulfonic or disulfonic acid compound comprises a sulfoacetic, sulfosuccinic, methanesulfonic, ethanedisulfonic, or hydroxybutane sulfonic, compound or mixtures thereof.

34. (new) A composite membrane as in Claim 27 wherein said organic sulfonic acid compound comprises methanesulfonic acid, trifluoromethanesulfonic acid or a mixture thereof.

35. (new) A composite membrane as in Claim 27 wherein said solution of an organic sulfonic acid compound comprises said organic sulfonic acid compound dispersed or dissolved in water, alcohol, glycol, alkoxy alcohol or a carboxylic acid or a mixture thereof.

REMARKS

The Applicants thank the Examiner for his time during the telephonic interview related to this case and his careful analysis of the claims. The Applicants have amended the claims as set forth above to eliminate the use of aryl sulfonic acids in the method of the preparation of membranes. A new set of claims (claims 27-35) have been added that parallel the original claims 15-20, 25 and 26, further containing the limitation that the membrane must have a flux of at least 15 gfd, rather than about at least 15 gfd. The Applicants submit that in view of the forgoing amendments and following arguments that the application is now in proper form for allowance.

Rejections under 35 U.S.C. 102(b)

The Examiner has rejected claims 15-20, 25 and 26 under 35 U.S.C. 102(b) as being anticipated by Chau (USP 4,983,291). An informal communication was filed with the Examiner on January 22, 2004 (copy enclosed as Appendix A, including references, to be entered into the case) presenting a number of arguments regarding the non-specificity of the teachings of Chau regarding sulfonic acids. The Applicants submitted that that one skilled in the art would not be motivated to select the specific classes of sulfonic acids listed in claim 15, specifically C_1 to C_6 alkyl, alkenyl, haloalkyl, haloalkenyl, or hydroxy sulfonic or disulfonic acids based on the teachings of Chau. The Examiner stated by telephone that the specific example of PTSA (see table 4) fell within the limitations of claim 15 and was sufficient to result in the rejection of the claim under 102(b). Therefore, the arguments presented by the Applicants regarding the non-specificity of the teachings of Chau were not relevant. The Applicants respectfully submit that the limitations of claim 15 were not previously met by the Chau reference, and with the limitation of aryl sulfonic acids from the claim, PTSA no longer falls within the limitations of the claim. Similarly, claim 27 is differentiated from Chau for the same reasons.

In his rejections, the Examiner pointed to the general teachings of Chau and suggested that the working examples contain examples of membranes that show a salt rejection of at least 25% and a flux of about at least 15 gfd under the conditions stated in the claims. In all of the working examples presented, the only example using a sulfonic acid was one teaching of the use of PTSA. The treatment resulted in a membrane with a flux of 13.57gfd when adjusted for the salt concentrations used in the claims (calculations were presented in the Informal Communication filed on October 15, 2003, copy enclosed as Appendix B to be entered into the case). No other specific data were presented on sulfonic acids.

On the top of page 5 of the informal communication sent to the Examiner on January 21, 2004, the Applicants submited that 13.57 gfd does not fall within the range of "about at least15 gfd" and request that the Examiner demonstrate that one skilled in the art would consider 13.57 gfd to fall within the range of about at least 15 gfd, noting that all of the data presented in Chau are to a tenth of a gfd. The Applicants have also added claim 27 which includes the limitations of claim 15, but instead recites "at least 15 gfd," rather than "at least about 15 gfd." Therefore, this claim clearly does not fall within the scope of Chau. The only membrane to meet the functional requirements of the claim were membranes treated with a solutions of 2 or 5% citric acid. These treatments resulted in a membrane with the required flux characteristics as claimed (21.8-19.0 gfd with a 2.1-3.5% salt passage under the conditions of claim 15; see table 2). However, citric acid is not a sulfonic acid, and the usefulness of citric acid in the preparation of membranes provides no suggestion for the use of sulfonic acids.

The Applicants have amended claim 15 to eliminate the use of aryl sulfonic acids. Claim 27 similarly does not recite the use of aryl sulfonic acids. Therefore, the specific example of PTSA does not fall within the limitations of the claim, as it meets neither the chemical nor the functional limitations required by the claim. Therefore, the rejection of claim 15 under 35 U.S.C. 102(b) is traversed and claim 27 is clearly distinguished from Chau. As

the remaining claims in the rejection are dependent on the now allowable claim 15, the rejection of claims 16-20, 23 and 25 are traversed. Similarly, claims 28-35 are not anticipated in view of the prior art.

Rejections under 35 U.S.C. 103(a) over Chau

In view of the traversal of the rejection of 102(b), the lack of a *prima facie* obviousness of the claims of the instant invention in view of the long list of compounds presented by Chau are now germane to the case. It is requested that the Examiner reconsider these arguments in view of the forgoing amendments to the claim.

Election of Species

The Applicants request that the Examiner consider the chart in the MPEP under section 2144.08(III). A flow chart is provided for the determination if a species or subclass is obvious in view of a genus. The Applicants submit that by the use of this chart, the selection of the specific subclass of the C_1-C_6 sulfonic acids claimed in this invention cannot be considered obvious. The text of the inquiries from the MPEP is in italics.

II.A.4.a. Is Genus So Small That Each Member is Inherently Disclosed?

No. Chau teaches a number of large classes of compounds. The Applicants submit that preparation of an exhaustive list of all of the compounds that would fall within the classes listed by Chau would be a substantial undertaking.

II.A. 4.b Are There Express Teachings That Would Have Motivated the Selection?

No. Many classes of compounds are taught. In order for the instant invention to be obvious in view of the Chau reference, one would first need to be motivated to select sulfonic acids, and then further select compounds that were not listed in the specification of Chau. The membranes that demonstrate the highest flux in Chau are those treated with citric acid, not with sulfonic acids. Sulfonic acids are substantially more expensive than citric acid. Organic

sulfonic acids, as required by the limitations of the claims of the instant application, require special handling and disposal. The only sulfonic acid specifically taught by Chau does not meet the functional limitations of the claim. There is no motivation to select sulfonic acids over the other classes taught, or to select the specific subgroup of sulfonic acids claimed in the instant invention. None of the compounds listed in claim 15 or 27 are specifically named in the Chau reference. No subclasses are defined in Chau based on the number of carbons or other factors.

II.A.4.c. Is there a teaching of structural similarity?

No. There is not substantial, overall structural similarity between the specific compounds claimed in the instant invention and the broad teachings of Chau. Morever, there is little structural similarity between the many classes of compounds taught by Chau that could suggest the use of low molecular weight organic, sulfonic acids. The only property that the compounds of Chau have in common is that they are all hygroscopic. If one were motivated by Chau to investigate the use of sulfonic acids for the treatment of membranes, there is essentially no size limitation on the compounds which can be substantially larger than the C_1-C_6 sulfonic acids of the instant invention. The Applicants submit that the commonality of a single functional group within acids does not constitute structural similarity sufficient to teach the small group of compounds claimed in the instant application.

II.A.4.d Is there any other teaching to support the selection of the species or subgenus?

No. The subgenus claimed is not taught by the prior art. The Applicants submit that if it were obvious to make the membranes of the instant invention in view of Chau, the membrane would have been made long ago as the Chau patent was issued in 1991. Therefore, the claim must be nonobvious under 103(a).

Rejections under 35 U.S.C. 103(a) over Chau in view of Koo

The Examiner has rejected claims 21, 23 and 25 as being unpatentable over Chau in view of Koo under 35 U.S.C. 103(a). The Examiner states that a case of prima facie obviousness may be made when the chemical compounds have very close structural similarities and similar utilities. The Examiner states that it would be obvious to one of ordinary skill in the art at the time of the invention to use the teachings of Koo in conjunction with the teachings of Chau to make polyamide reverse osmosis membranes, because Koo teaches that these sulfonic acids are equivalent for providing high flux, high rejection membranes. The Examiner has pointed to three of the many sulfonic acids listed in Koo, specifically methane sulfonic acid, ethane sulfonic acid and benzene sulfonic acid, and has stated that it would be obvious to use these acids in the context of Chau. Again, the Applicants submit that there are no teachings in Koo to suggest the use of these sulfonic acids of the many sulfonic acids listed. These arguments are presented in the paragraph bridging pages 8 and 9 in the communication of January 22, 2004.

The Applicants further submit that the use of the strong acids, including sulfonic acids, in Koo is not the same as the use of sulfonic acids in Chau; therefore, there is no motivation to combine the references. The sulfonic acids in Koo are used as a reactant to make a salt with a polyfunctional amine to make the membrane, not as a post-treatment for a completed membrane. The Summary of the Invention of Koo states (col 2, ln 64ff):

> According to one aspect of the present invention, there is disclosed a polyamide membrane, said polyamide membrane comprising a reaction product of (i) a polyfunctional amine and (ii) an amine selective reactant selected from the group consisting of a polyfunctional acyl halide, a polyfunctional sulfonyl halide and a polyfunctional isocyanate (iii) in the presence of a salt-containing compound, said salt containing compound being a reaction product of a strong acid and a polyfunctional amine. [emphasis added]

In Koo, the strong acid is used to make a salt, not to treat a membrane.

Sulfonic acid is used in Chau to treat membranes. The Summary of the Invention of

Chau (col 3, ln 36-50) reads:

> In one aspect an embodiment of this invention resides in a high flux semipermeable membrane prepared by coating a porous support backing material with an aqueous solution of a polyamine, removing excess solution, contacting the coated porous support backing material with an organic solvent solution of a polyacyl halide, polysulfonyl halide or polyisocyanate to form a reaction product within and/or on the surface of said porous support backing material, treating the resultant composite with a solution of an acid [emphasis added] or amine salt selected from the group consisting of hydroxypolycarboxylic acids, polyaminoalkylene polycarboxylic acids, sulfonic acids, amino acids, amino acid salts, amino salts of acids, polymeric acids and inorganic acids, drying and recovering the resultant high flux semipermeable membrane.

Koo uses a "salt containing compound" that is the reaction product of a strong acid and a polyfunctional amine to make membranes. The salt containing compound is a reaction product of a strong acid and a polyfunctional amine. Koo does not use strong acids directly in the preparation of membranes. Koo does not use strong acids in the treatment of membranes.

Chau teaches the treatment of membranes with any of a number of compounds including sulfonic acids to treat membranes to allow to "enable the membrane to be stored in a dry manner prior thereof to use in separation process." (abstract) The Applicants submit that one of ordinary skill in the art would not consider the preparation of a salt and the treatment of membranes to be similar utilities. In Koo, the acid is never used directly with the membrane. If the salt and the acid were functionally similar, Koo would not be motivated to make the salt. Clearly this is not the case. Therefore, the use of the sulfonic acid in Chau and Koo cannot be considered similar. Therefore, the combination of references is improper and the rejection is traversed. The Applicants submit that claims 21, 23 and 25 should now be allowable, and the rejection under 35 USC 103(a) is traversed.

Secondary Considerations

A rejection under 35 U.S.C. 103 must take into consideration the fundamental inquiries set forth in *Graham v. John Deere* including the scope and content of the prior art, the difference between the prior art and the claims at issue and the level of ordinary skill in the pertinent art at the time the invention is made. In fields such as chemistry where accomplishments tend to be incremental, it can be difficult to avoid hindsight and determine what might have been obvious to one of ordinary skill in the art at the time of filing. Invention does not require a flash of genius, but instead simply a useful advance. To provide better perspective on what would have been obvious, secondary considerations may be used to determine the non-obviousness of the invention. These considerations include, but are not limited to:

(a) commercial success;

(b) long felt, but unresolved need;

(c) failure of others;

(d) recognition of problem;

(e) failed attempts to solve problem;

(f) simultaneous, independent invention by others;

(g) competitors prompt copying;

(h) acclaim by others;

(i) licensing of patent to industry;

(j) teaching away by those skilled in the art; and

(k) results of invention unexpected by skilled.

Secondary considerations were found to be critically important in the Federal Circuit case of *Ecolochem, Inc v. Southern California Edison Company*. Ecolochem sued Southern California Edison for infringement of its patent (USP 4,818,411, hereafter the '411 patent). Southern California Edison responded by saying that the patent was invalid as it was obvious

in view of the prior art. The court relied heavily on secondary considerations to provide a view of what was obvious to one skilled in the art at the time of the invention and found that the Ecolochem patent was non-obvious in view of the prior art despite the fact that it was a combination of methods that were all well known at the time of the filing of the application. Southern California Edison was found guilty of willful infringement.

The Ecolochem '411 patent taught the combination of a number of methods that were all well known. At the time of the filing of the application, the use of hydrazine to remove dissolved oxygen from water was well known. The reaction was known to be slow at room temperature. Activated carbon was known to be useful as a catalyst. However, the use of activated carbon results in water with an unacceptably high level of contaminants in the water for use in nuclear power plants. It was discovered by the inventors that the use of ion exchange resins, well known at the time of the application, allowed for the removal of carbon contaminants and the unreacted hydrazine, resulting in water that could be used for the desired application. Therefore, although the individual steps were not novel, their combination was found to be novel and non-obvious in view of the prior art. Therefore, a patent was issued. The invention was well received by those in the industry and resulted in substantial commercial success for Ecolochem, Inc. Ecolochem made sales to over 25 power plants and earned over $10 million in revenue in its first seven years. These factors were considered by the court to be significant in the determination that the patent had been properly granted and that the claims were, in fact, novel and non-obvious in view of the prior art.

The Applicants submit that the Ecolochem v. Southern California Edison case is relevant to this case. Treatment of filtration membranes is known to those skilled in the art. Sulfonic acids, including those specifically claimed in this application, are well known to those skilled in the art. However, the treatment of filtration membranes with the sulfonic acids specified by the claims of the invention results in a membranes that are superior to other commercially available membranes, resulting in commercial success of the membrane and

acclaim from those in the industry.

The Applicants have enclosed three documents to support the non-obviousness of the instant invention based on secondary considerations. These documents demonstrate commercial success of Hydranautics based on the membranes of the invention. This success was due to an unmet need in the industry and the failure of others to provide membranes with the appropriate functional characteristics. The commercial success of Hydranautics has been coupled with acclaim from those in the industry who have independently tested the membranes of the invention against other commercially available membranes and have found them to be superior.

The membranes of the instant application have achieved substantial commercial success despite their higher direct cost when compared to other commercially available membranes designed for a similar use. The Applicants have enclosed three published manuscripts, each demonstrating the advantages of their membranes as determined by others in the industry.

The Applicants first wish to direct the Examiner's attention to the paper entitled "Optimizing the Performance of Low Fouling Membranes for the World's Largest Nanofiltration Plant" by Kiefer et al, presented at the American Water Works Association 2003 Annual Conference, March 2-5. It should be noted that the authors of the paper are not affiliated with Hydranautics and that the data in the paper were obtained independent of Hydranautics. The membrane of the instant invention is designated at the ESNA LF membrane in the paper. The manuscript details the analysis performed by the City of Boca Raton, Florida to determine what membranes would be used in their new water purification plant. After an extensive analysis, and much to the surprise of those performing the analysis, the Hydranautics membranes were selected.

The details of the testing are provided in the figures. Figure 1 is a graph of Mass Transfer Coefficient vs. Time of Operation for three commercially available membranes, not

including the membrane of the instant invention. None of the membranes were found to be acceptable despite the use of a variety of combinations of acids and antiscalants (paragraph immediately preceding Figure 1). Figure 2 shows a surprising improvement seen with the commercially available membranes without pretreatment with acids or antiscalants; however, the mass transfer coefficient was not as high as would have been preferred. Therefore, other sources of membranes were sought out for testing.

A prototype membrane from Hydranautics was tested (page 4, new low fouling membrane). The data are presented in Figure 3 and show better performance than the membranes tested in Figure 2. In the text it is noted that the prototype Hydranautics membranes were stable for six to eight months without the addition of acid or antiscalant.

Side by side testing of the Hydranautics membrane for calcium passage against three other commercially available membranes is shown in Figure 4. Figure 5 is again a graph of mass transfer coefficient over time comparing the Hydranautics membrane to the best performing membrane in the previous test. Again, the Hydranautics membrane outperformed the other commercially available membrane.

A summary of the advantages of the Hydranautics membrane are presented in the bullet points in the middle of page 10. However, the strongest demonstration of the uniqueness of the membranes of the instant invention is shown in the first full paragraph on page 10. The membranes of Hydranautics were not initially included in the testing due to capital cost considerations. However, none of the commercially available membranes were able to function at a sufficient level to meet the requirements of the plant. Therefore, the Hydranautics membranes were finally selected for their outstanding performance, and overall cost savings due to this high level of performance. The City of Boca Raton placed an order for 8316 elements for a total cost of $4.2 million.

The paper "Optimizing New Low Fouling Nanofiltration Membrane Performance for Deerfield Beach" by Kiefer et al. (presented at the American Water Works Association 2003

Annual Conference, March 2-5) presents another location wherein the ESNA LF membranes of Hydranautics were demonstrated to function at a substantially superior level to other commercially available membranes in a hybrid system. Again, the manuscript was not written by an employee of Hydranautics demonstrating the enthusiasm with which the membranes were accepted by those in the industry. In a first direct comparison, the ESNA LF membrane was shown to be far more stable than the two membranes tested for a hybrid water purification system (see Figure 3), demonstrating the failure of others to produce a membrane with the necessary characteristics. The water product generated by the Hydranautics single membrane process fell slightly outside the parameters required by the Deerfield Beach plant, so a hybrid membrane system was developed using the membranes of the instant invention. Again, due to the high quality of the membranes of the instant invention, the higher cost of the membranes was negated by the substantially improved performance. The Deerfield Beach system is further discussed in the manuscript entitled "Advantages of New Low Fouling Nanofiltration Membranes vs. a Hybrid Membrane Design for Deerfield Beach" by Kiefer and Jackson presented at the American Water Works Association 2002 Annual Conference, June 16-20. The City of Deerfield Beach placed an order for 2016 elements for a total cost of $900,000.

The Applicants submit that the attached references demonstrate the non-obviousness of any method to make membranes that meet the requirements of the claims of the instant application. It is not known by the Applicants what methods were used for the preparation of the membranes tested in the attached references.

Commercial success has clearly been demonstrated. An unmet need in the industry has been clearly demonstrated. The failure of others to meet the need has been clearly demonstrated. Acclaim by those in the industry has been clearly demonstrated. A prototype membrane from Hydranautics was tested as none of the commercial membranes tested were able to meet the needs of the plants. The publication of the papers by others, not by

Hydranautics, demonstrates that they were well received by the industry and acclaimed for their benefits. As stated above, if the results of the treatment of the membranes was obvious, it would have been done long ago as the Chau patent was issued in 1991. Therefore, many of the secondary considerations have been satisfied regarding the novelty and non-obviousness of the instant invention. Therefore the rejection of the claims is overcome and the new claims are neither anticipated nor obvious in view of the prior art.

FEES

It is believed that no fee is due with this response. However, if a fee is due, the Commissioner is hereby authorized to charge Deposit Account 50-1990 referencing case number 7703-PA02.

CONCLUSIONS

Respectfully submitted,

Dated: _____

By: _____
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